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Report 2488

Failure Mechanism of Urethane Elastomer Coated Fabric Collapsible Fuel Tanks

Houry O. Fauer, Jr.

Authored By: Saul Touchet

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#### PREFACE

This report details testing of polyurethane materials currently being used or considered for use in collapsible bulk storage fuel tanks. This work is in support of the Fuels and Water Supply Division, Logistics Equipment Directorate, Belvoir Research, Development and Engineering (RD&E) Center and was performed by the Rubber and Coated Fabrics Research Group, Materials, Fuels and Lubricants Laboratory, Belvoir RD&E Center.

The US Army currently has a large number of fabric reinforced elastomer tanks ranging in size from 100 to 210,000 gallons used for the storage of fuels and other liquids in the field. Figures 1 and 2 show a 5,000 barrel collapsible fuel tank undergoing testing in Panama. Although this tank is already displaying signs of weathering and deterioration, it has not failed. Collapsible fuel tanks may be required to hold hydrocarbon fuels such as JPs, diesel, and gasoline for long periods of time under adverse conditions. The elastomer usually used as the coating material on these tanks is a polyester urethane. Polyester urethanes are inherently resistant to fuels and abrasion, but they are susceptible to attack by water (hydrolysis) and ultraviolet radiation. Protective ingredients are added to these urethanes to protect them from those types of deterioration.

The coated fabric materials used in these collapsible fuel tanks are tested to conform to military specifications, but in field use they have prematurely failed. These failures are frequent in Panama with tanks holding diesel fuel. Figures 3 and 4 show a seam on a collapsible fuel tank that failed during recent testing in Panama. This type of failure is common and is usually caused by deterioration of the coating and resultant delarnination between the elastomer coating and the fabric, and finally seam failure. The delamination is a result of elastomer deterioration and loss of inherent properties. Although research work has been performed to determine what was happening to the diesel fuel to cause the elastomer's accelerated deterioration, no progress has been made to date.

The reason for the collapsible fuel tank failures is due to diesel fuel extracting out the protective ingredients in the polymer, allowing hydrolysis to occur and thus shortening the tank's expected service life. This report details testing performed to prove the extraction idea. All figures referenced in this report are shown in the appendix.



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### SECTION I. POLYURETHANE ELASTOMERS

Polyurethane elastomers cover a wide range of applications due to their ability to be modified for a given set of end-use conditions. These modifications are done during the formation of the urethane by varying the types of monomers or initial ingredients. All urethanes are made by different monomer blocks or segments that are linked together to form the backbone of the urethane elastomer. The common block in urethane elastomers is the urethane link as shown in Figure 5. The other segments attached to this link give the urethane its specific properties. The main types of segments linked to the urethane block are esters and ethers. *Ether* urethanes are used in applications where resistance to hydrolysis is important but fuel resistance is not. *Ester* urethanes are used for their hydrocarbon fuel resistance but are susceptible to hydrolytic attack.

When an ester urethane is hydrolytically attacked, the bond between the ester and urethane block is broken, decreasing the urethane's strength and eventually causing the urethane to become brittle or tacky. In the case of collapsible fuel tanks, the urethane will delaminate from the fabric. Additives can be added to slow down the deterioration process caused by water. A literature search revealed that only one type of hydrolytic stabilizer is used in ester urethanes. This material is an aromatic polycarbodiimide, which was used in the urethane coatings when collapsible tanks for Army fuel storage were fabricated.

#### SECTION II. INVESTIGATION

#### SCOPE

The purpose of this work was to determine if an extractive process was taking place with the urethane elastomer used on coated fabric bulk fuel collapsible tanks. The elastomer's degradation was monitored by the degree of deterioration of the elastomer's physical properties.

#### **Preliminary Testing**

To determine if an extraction type process was occurring, the first test was to determine if the protective ingredient was soluble in fuels. Small samples of aromatic polycarbodiimide were placed in vials containing five different solvents: water, acetone, JP-8, and two types of diesel fuel. The aromatic polycarbodiimide completely dissolved in both diesel fuels and in the JP-8, and was slightly soluble in the acetone. It was completely insoluble in water. In the polymer, this chemical would not be bonded to the elastomer, allowing the fuel to dissolve and remove it from the coating.

The ensuing test involved small pieces of coating from a collapsible fuel tank. Several pieces of the coating were placed in diesel fuel for 2 weeks, allowing the fuel to remove the protective

ingredients. The samples were then placed in flasks containing water and aged in an oven at 180°F. Samples of the coating that were not extracted were also tested in water at 180°F for control purposes. Both samples were checked daily for signs of deterioration by removing the sample from the water and using a thumbnail to test their toughness. Although rot a standard procedure, this was done only to ascertain how fast the deterioration was taking place. After only 7 days, the extracted samples showed signs of degradation in that they were much softer than the control samples. At 21 days in water, the extracted samples crumbled when tested. The control samples did not show any signs of degradation until 28 days of immersion when they showed some degree of softening.

These preliminary tests were used only to determine if the extraction process was occurring. The results from this testing indicated that it was occurring and a more controlled test should be performed.

#### **Primary Testing**

#### Materials

Four ester polyurethane compounds were tested (see Table 1) since only ester compounds were on the fabrics which failed in the field. The first three compounds were virgin, uncompounded materials currently used for a laboratory database. They were supplied in sheet form from which the samples were prepared. The fourth compound was a coating compound currently used as a coating on fabrics for collapsible fuel tanks. This material had been compounded and contained reinforcing fillers as well as additives such as hydrolytic and ultraviolet (UV) stabilizers. It was also supplied in sheet form without the fabric reinforcement.

#### Table 1. List of Polyurethane Compounds Tested

PU-1	An uncompounded urethane with an ester backbone; does not have any protective ingredients such as hydrolytic stabilizers.
PU-2	An uncompounded $\psi$ ethane with an ester backbone; has a hydrolytic stabilizer added for water resistance.
PU-3	An uncompounded urethane with an ester backbone; has more hydroltyic stabilizers than PU-2 for increased water resistance.
PU-4	A compounded urethane used as a coating on fabrics for collapsible fuel tanks; has protective ingredients added for protection from hydrolysis and UV.

#### Fuels

Two test fuels were used, a diesel fuel and an unleaded gasoline. These fuels were on hand and represented fuels in the field. Diesel fuel was used since it is the fuel most frequently associated with premature collapsible fuel tank failures. Gasoline was used since it often causes the worst deterioration in laboratory testing.

#### **PREPARATION**

#### Sample Preparation

Test specimens were prepared from the supplied urethane sheets in accordance with ASTM D-412, Rubber Properties in Tension, using a Die-C Dumbbell. The samples were cut and original thicknesses were measured. Sets of three samples of each elastomer were prepared for differing time intervals and test conditions. Nine test conditions were used and are listed in Table 2. Original samples were also tested. The time intervals used for water aging were 14 days, 28 days, and 42 days. The test temperatures were room temperature (72°F± 4°F) and the temperature of 160°F± 5°F was done in a forced air oven.

#### Table 2. Test Conditions Samples Aged Under

- 1 Aged in water at 160°F.
- 2 Aged in diesel fuel at room temperature.
- 3 Extracted in diesel fuel at room temperature for 14 days, then immersed in water at 160°F for times indicated.
- 4 Aged in diesel fuel at 160°F.
- 5 Extracted in diesel fuel at 160°F for 14 days, then immersed in water at 160°F for times indicated.
- 6 Aged in gasoline at room temperature.
- 7 Extracted in gasoline at room temperature for 14 days, then immersed in water at 160°F for times indicated.
- Aged in gasoline at 160°F.
- 9 Extracted in gasoline at 160°F for 14 days, then immersed in water at 160°F for times indicated.

#### **Procedures**

The immersion of the elastomer materials and the determination of the effects on their properties were performed in accordance with ASTM D-471, Rubber Property-Effect of Liquids with the exception of measurements for the determination of the volume change caused by the immersion in the test fluid. The three test specimens were placed in test tubes with screw closures to prevent evaporation and were tested as stated above.

#### **RESULT DATA**

The results reported were produced by the average of three specimens. The physical properties of all the elastomeric materials are tabulated in Tables 3 through 6 and graphically represented in Figures 6 through 21 (see Appendix).

Table 3. Tensile Strength Data

Conditions	Original	14 Days	28 Days	42 Days
1	3860	1630	490	Α
2	3860	4590	2850	3320
3	3860	1720	530	Α
4	3860	3700	2780	1560
5	3850	1030	310	Α
6	3860	4190	3670	3580
7	3860	1430	480	Α
8	3860	3220	1220	2670
9	3860	1200	390	Α
		PU-2		
1	2880	2430	1480	700
2	2880	2860	2760	2750
3	2880	2170	1160	660
4	2880	2640	2360	1950
5	2886	1840	780	430
6	2880	2490	2760	2720
7	2880	2290	1530	760
8	2880	2720	1990	2430
9	2880	1940	1200	630
		PU-3		
1	3930	3380	2700	1620
2	3930	3720	347	4170
3	3930	3130	2670	1610
4	3930	3500	3120	2880
5	3930	2830	1770	1070
6	3930	3910	3790	3310
7	3930	2920	2780	1710
8	3930	3290	3130	2540
9	3930	2790	2020	1210
		PU-4		
1	5150	4350	4300	3750
2	5150	6190	5400	4830
3	5150	4620	3910	3330
4	5150	5100	4590	3860
5	5150	4680	3330	1450
6	5150	4420	4760	4490
7	5150	4030	3410	2790
8	5150	3750	4800	3500
9	5150	3440	2700	940

Table 4. Ultimate Elongation Data

2 480 600 480 480 3 480 700 170 A 4 480 740 770 780 5 480 640 90 A 6 480 680 580 570 7 480 700 180 A	Α
2 480 600 480 480 3 480 700 170 A 4 480 740 770 780 5 480 640 90 A 6 480 680 580 570 7 480 700 180 A	80 <b>A</b>
3     480     700     170     A       4     480     740     770     780       5     480     640     90     A       6     480     680     580     570       7     480     700     180     A	Α
4     480     740     770     780       5     480     640     90     A       6     480     680     580     570       7     480     700     180     A	
5     480     640     90     A       6     480     680     580     570       7     480     700     180     A	
6 480 680 580 570 7 480 700 180 A	A
7 480 700 180 A	
	Α
8 480 830 820 840	
	Α
PU-2	
1 490 670 630 210	10
2 490 520 510 480	
3 490 670 600 260	
4 490 590 620 650	
5 490 760 390 80	
6 690 530 570 540	
7 490 680 680 340	
8 490 680 720 690	
9 490 680 610 170	
PU-3	
1 450 510 510 420	20
2 450 460 470 470	
3 450 520 530 430	
4 450 470 520 500	
5 450 510 420 150	
6 450 510 510 440	
7 450 520 560 450	
8 450 500 530 530	
9 450 510 500 270	
PU-4	
1 500 580 560 570	70
2 500 500 540 530	
3 500 580 580 580	
4 500 570 560 570	
5 500 600 620 460	
6 500 580 540 580	
7 500 610 590 600	
8 500 610 580 610	
9 500 630 630 190	

Table 5. Tensile Strength Retention Data

Conditions	14 Days	28 Days	42 Days
1	42.2%	12.7%	0.0%
2	118.9%	73.8%	86.0%
3	44.6%	13.7%	C.0%
	95.9%	72.0%	40.4%
4 5 8	26.7%	8.0%	0.0%
8	108.5%	95.1%	92.7%
7	37.0%	12.4%	0.0%
8	83.4%	31.6%	69.2%
9	31.1%	10.1%	0.0%
		PU-2	
1	84.4%	51.4%	24.3%
2	99.3%	95.8%	95.5%
3	75.3%	40.3%	22.9%
4	91.7%	81.9%	67.7%
5	63.9%	27.1%	14.9%
5 6	86.5%	95.8%	94.4%
7	79.5%	53.1%	26.4%
8	94.4%	69.1%	84.4%
9	67.4%	41.7%	21.9%
		PU-3	
1	86.0%	68.7%	41.2%
	94.7%	88.3%	106.1%
2 3	79.6%	67.9%	41.0%
4	89.1%	79.4%	73.3%
5	72.0%	45.0%	27.2%
5 6	99.5%	96.4%	84.2%
7	74.3%	70.7%	43.5%
8	83.7%	79.6%	64.6%
9	71.0%	51.4%	30.8%
		PU-4	
1	84.5%	83.5%	72.8%
2	120.2%	104.9%	93.8%
3	89.7%	75.9%	64.7%
4	99.0%	89.1%	75.0%
5	90.9%	64.7%	28.2%
6	85.8%	92.4%	87.2%
7	78.3%	66.2%	54.2%
8	72.8%	93.2%	68.0%
9	66.8%	52.4%	18.3%

Table 6. Ultimate Elongation Retention Data

Conditions	14 Days	28 Days	42 Days
1	137.5%	29.2%	0.0%
2	125.0%	100.0%	100.0%
3	145.8%	35.4%	0.0%
4	154.2%	160.4%	162.5%
5	133.3%	18.8%	0.0%
5 6	141.7%	120.8%	118.8%
7	145.8%	37.5%	0.0%
8	172.9%	170.8%	175.0%
9	152.1%	22.9%	0.0%
		PU-2	
1	136.7%	128.6%	42.9%
	106.1%	104.1%	98.0%
2 3	136.7%	122.4%	53.1%
	120.4%	126.5%	132.7%
, 5	155.1%	79.6%	16.3%
4 5 6 7	108.2%	116.3%	110.2%
7	138.8%	138.8%	69.4%
8	138.8%	146.9%	140.8%
9	138.8%	124.5%	34.7%
		PU-3	
•	113.3%	113.3%	93.3%
1	102.2%	104.4%	104.4%
2	115.6%	117.8%	95.6%
2 3 4	104.4%	115.6%	111.1%
4	113.3%	93.3%	33.3%
5 6	113.3%	113.3%	97.8%
7	115.6%	124.4%	100.0%
	111.1%	117.8%	117.8%
8 9	113.3%	111.1%	60.0%
		PU-4	
1	116.0%	112.0%	114.0%
2	100.0%	108.0%	106.0%
3	116.0%	116.0%	116.0%
4	114.0%	112.0%	114.0%
	120.0%	124.0%	92.0%
5 6	116.0%	108.0%	116.0%
7	122.0%	118.0%	120.0%
	122.0%	116.0%	122.0%
8		126.0%	38.0%
9	126.0%	140.0%	30.0%

#### SECTION III. TEST RESULTS

#### TEST DESCRIPTIONS

#### **Tensile Strength**

Tensile strength is a standard control measurement used in the rubber industry. It is useful for comparing compounds for quality control, and as a test to determine the extent an elastomer has deteriorated when exposed to a hostile environment. In most cases, if an elastomer has a large reduction in tensile strength after immersion in a test fluid, a relatively short lifespan can be expected. Conversely, a very small loss in tensile strength indicates a longer lifespan.

The tensile strength of the elastomeric materials tested showed some variation. Compounds PU-1, PU-2, and PU-3 were virgin, unreinforced materials, while PU-4 had a filler for reinforcement. This was evident in the original tensile strengths. PU-4 had the highest original tensile strength of the four materials tested.

With a few exceptions, the tensile strength data gathered showed a decreasing trend in the tensile strength with immersion time. Some samples showed a slight increase during the first test period but by the end of the testing, all samples (with one exception) showed at least some decrease in tensile strength from the original values.

The tensile strength data is tabulated in Table 3 and shown graphically in Figures 6 through 9 (see Appendix).

#### **Ultimate Elongation**

Elongation is defined as the increase in length expressed as a percentage of the initial gage length. The ultimate elongation is the length reached when the sample breaks. Elongation measurements are used in much the same way as tensile strength data; however, a large increase or decrease in elongation may represent deterioration.

The test results showed primarily increases in elongation during the initial testing periods and, in most cases, decreases by the end of the testing periods. The elongation frequently increases during the initial time periods for test samples, due to the fluid acting as a plasticizer. After time, the deterioration processes cause a weakening of the elastomer's backbone chain which effects the elongation.

The test results for the elongation are tabulated in Table 4 and shown graphically in Figures 10 through 13 (see Appendix).

#### Tensile Strength Retention

Tensile strength retention is the measured tensile strength after aging, divided by the original tensile strength. This calculation usually takes into account changes in the elastomer due to volume changes but, in this testing, no volume change data was collected. The tensile strength retention data often shows trends in the tensile strength more clearly. The tensile strength retention values are usually expressed as a percentage.

The calculated tensile strength retention values are tabulated in Table 5 and shown graphically in Figures 14 through 17 (see Appendix).

#### **Elongation Retention**

Elongation retention is the measured elongation after aging divided by the original elongation. The volume swell is not a factor in these calculations. The elongation retention, expressed as a percentage, often helps define trends in elongation more clearly than the elongation data alone.

The calculated elongation retention values are tabulated in Table 6 and shown graphically in Figures 18 through 21 (see Appendix).

#### **ELASTOMER TESTING RESULTS**

#### PU-1

This polyurethane is based on an ester linkage in the backbone. It is uncompounded and does not contain any fillers or protective ingredients. It is used to get an idea of what reactions take place with just the base elastomer; specifically, without stabilizers during testing.

The test results confirmed that PU-1 is an ester-type polyurethane. The tensile strength test results for PU-1 are shown in Table 3 and in Figure 6. The tensile strength showed marked decreases in the samples that were aged in water (conditions 1, 3, 5, 7, and 9). None of the samples that were aged in water had enough integrity to be tested after 28 days. There was some fluctuation in the tensile strength values for the fuel aged samples (conditions 2, 6, and 8). (This fluctuation is not uncommon in testing urethane elastomers.) The reduction in tensile strength was due to the fuel immersion but, at an equilibrium time, the value will reach a minimum. After this equilibrium point, the urethane's tensile strength increases to a higher value before deterioration effects cause the tensile values to decrease and eventually fail.

The ultimate elongation results in Table 4 and Figure 10 showed that the material degraded severely under the water aged conditions 1, 3, 5, 7, and 9. The samples did not have enough integrity to be tested at 42 days under the aforementioned conditions. The samples showed degradation under fuel aged conditions 4, 6, and 8 but did not fail. The increases in ultimate elongation in conditions 4, 6, and 8 showed that slight detrimental changes occurred in those samples and there was some deterioration in the sample aged under condition 2.

The tensile retention results shown in Table 5 and in Figure 14 did not display any trends that were not evident in the tensile strength results. This was not surprising in that the tensile strength results clearly showed degradation trends for the test samples.

The elongation retention data in Table 6 and in Figure 18 displayed the same results as the ultimate elongation results. Like the tensile retention results, this was not surprising because of the clear-cut nature of the ultimate elongation results.

The test data showed the expected trends for an ester polyurethane in that the material maintained most of its integrity in fuel but degraded when exposed to water. This material as is would not be suitable for use as a coating on coated fabric fuel storage tanks. It should be noted, however, that this material did not have any ingredients added to it to protect it from hydrolytic attack.

#### PU-2

The PU-2 urethane is based on an ester linkage in the backbone and is uncompounded with the exception of a small amount of hydrolytic stabilizer. This material is similar to the PU-1 compound with the exception of the hydrolytic stabilizer. The PU-2 material was used as a comparison to the PU-1 material to determine what effects a hydrolytic stabilizer would have.

The test results for PU-2 were similar to the results of PU-1; however, the samples did not fail under any conditions. The tensile strength results are listed in Table 3 and shown in Figure 7. The tensile strength showed a marked decrease for those samples aged in water (conditions 1, 3, 5, 7, and 9). None of the samples failed to a point that they could not be tested. The samples extracted in fuel and then aged in water showed a greater decrease in the tensile strength after 42 days than the samples aged in just water. The samples extracted in fuels only (conditions 2, 4, 6, and 8) showed some fluctuation in values as explained earlier. The samples aged in diesel fuel at 160°F (condition 3) showed a consistent loss in tensile strength similar to the samples that were water aged but did not deteriorate nearly as much.

The elongation results for PU-2 are listed in Table 4 and shown in Figure 11. The samples that were aged in water (conditions 1, 3, 5, 7, and 9) showed increases in the elongation for 14 days and, in most cases 28 days, then a large decrease by 42 days. The changes in the elongation were due to the equilibrium effects of urethanes as previously stated. The large variations in the elongation showed

that deterioration was occurring. The elongation for the samples aged in fuels at room temperature (conditions 2 and 6) showed slight changes in ultimate elongation, while the samples aged in fuel at 160°F (conditions 4 and 8) showed larger changes in elongation. These increases in elongation were due to the fuels acting as plasticizers in the urethane. A plasticizer allows greater elongation with a slight loss of tensile strength. This is evident if the tensile strength results for conditions 2, 4, 6, and 8 are reviewed.

The tensile retention results are listed in Table 5 and shown in Figure 15. The apprarent trends in the tensile strength data were duplicated in the tensile retention data. The volume change information (not collected during this testing) may have shifted some of the tensile retention data, especially the samples aged in fuel.

The ultimate elongation information is listed in Table 6 and shown in Figure 19. As with the tensile strength retention values, the information gathered from the ultimate elongation retention results duplicated the ultimate elongation tendencies.

The test data for PU-2 showed similarities with the PU-1 information in respect to the rapid deterioration of the samples that were aged in water (conditions 1, 3, 5, 7, and 9). The difference was the hydrolytic stabilizer added to PU-2 which allowed those samples to last longer in the water (42 days for the PU-2 samples as opposed to only 28 days for the PU-1 samples). The fluctuations in the data, particularly both types of ultimate elongation data, is a characteristic phenomena of polyurethanes aged in fuels. The equilibrating effect has not been explored but believed to be caused by the fuel acting in a plasticizing role. The fuel will cause the plasticizers (if any) to be removed and the fuel will replace the plasticizer in the polymer. While this transfer is taking place, there could be some variation in properties.

#### PU-3

The PU-3 urethane is also based on an ester linkage in the backbone and is uncompounded with the exception of the hydrolytic stabilizer. This compound contains more stabilizer than PU-2, giving greater water resistance. This material was provided for evaluation and was tested because of its similarity to PU-2.

The results from the tensile strength testing are listed in Table 3 and shown in Figure 8. Again, as expected with ester-based urethanes, the samples aged in water (conditions 1, 3, 5, 7, and 9) showed the greatest amount of degradation, with conditions 5 and 9 the worst. The fluctuation in condition 2 was explained earlier and the other samples aged in fuel only showed some deterioration. The hydrolytic stabilizer prevented any of the samples from failing completely in the water testing.

The ultimate elongation results listed in Table 4 and shown in Figure 12 represent the equilibrating effect seen in urethanes. Although there were some typical fluctuations in the elongations, there was only marginal deterioration observed with the exception of samples run under conditions 5 and 9. These conditions had the samples aged in diesel fuel and gasoline, respectively, at an elevated temperature of 160°F, then aged in water. The extraction process would work better at a higher temperature because it would allow faster removal of the stabilizers. This was evident by the large decrease in ultimate elongation by 42 days and the lowest tensile strength values observed for conditions 5 and 9.

The tensile strength retention information is listed in Table 5 and shown Figure 16. The data showed trends in the aging similar to those observed for the tensile strength. The loss of tensile strength for conditions 5 and 9 are evident in the graph (Figure 16). Both conditions showed drops in the tensile strength to 30% or less of the original tensile strength, while the other samples aged in water (conditions 1, 3, and 7) showed losses of up to 40%.

The ultimate elongation data listed in Table 6 and shown in Figure 20 showed the same information determined from the previous observations concerning PU-3. Again, the large decreases in elongation are evident only in those samples extracted at an elevated temperature, then aged in water.

The results from PU-3 showed that the most deleterious conditions for aging urethanes were conditions 5 and 9. Those conditions extracted the urethane in fuels at elevated temperatures before exposing the samples to water. The additional hydrolytic stabilizers were not removed as quickly during extraction in fuels at room temperature allowed the samples tested under conditions 3 and 7 more integrity. The samples ager in just water underwent less deterioration also due to the increase of hydrolytic stabilizer. This information is important for developing accelerated aging tests.

In comparing PU-3 to PU-2, it was apparent from the tensile retention and pltimate elongation information that the additional hydrolytic stabilizer gave better protection in PU-3. Although the original values for the tensile strength were different, the tensile retention showed less strength lost during water aged testing (conditions 1, 3, 5, 7, and 9). The same can be said for the elongation values. The retention values for tensile strength and ultimate elongation were higher for PU-3 than PU-2.

#### PU-4

The PU-4 urethane compound is an actual coating compound used on collapsible fuel tanks. It is based on an ester backbone and is fully compounded; that is, it contains protective ingredients, fillers, and other ingredients to assist processing. The information gathered from this material is the most useful because it is not an experiemental material as PU-1, PU-2, and PU-3; it is an actual material used in the field and may have been on a tank that prematurely failed.

The tensile strength data for PU-4 is listed in Table 3 and shown in Figure 9. The information derived from the data for PU-4 showed similarities to the previous urethane materials. To water aged conditions 1, 3, 5, 7, and 9 showed the greatest loss in tensile strength for the material, although some loss in tensile strength is recorded for the samples in conditions 2, 4, 6, and 8. The fluctuations, as explained previously, are evident in this data; most notably, conditions 2, 6, and 8. The samples that were extracted in fuels at an elevated temperature and then exposed to water showed the greatest losses in tensile strength. This was due to the same process which occurred to the PU-3 material.

The results for the ultimate elongation are listed in Table 4 and shown in Figure 13. The fluctuation phenomena is very apparent in those results. The fact that this was a fully compounded material enhances the fluctuations due to the larger amounts of likely used plasticizers and processing aids. As the fuel replaced some of the chemicals in the elastomer, some varying changes occurred. It is noteworthy to point out that these fluctuations were not nearly so apparent in the tensile strength results. The elongation showed some slight deterioration for all the testing conditions, but most notably conditions 5 and 9. Under those two conditions, the elastomer's elongation eventually went below the original value. This coincided with the observations made concerning the tensile strength. It was under the same conditions, extraction at an elevated temperature, then exposure to water that the largest tensile strength losses occurred. This again was very similar to what happened to material PU-3.

The tensile strength retention data is listed in Table 5 and shown in Figure 17. The results emphasized the large loss in strength for samples under conditions 5 and 9. The tensile strength retention was less than 30% of the original value at 42 days for condition 5 and 9. All the samples lost some of their tensile strength as compared to the original measurement.

The ultimate elongation retention values listed in Table 6 and shown in Figure 21 showed the same information as the ultimate elongation values. The previously discussed fluctuations are evident as well as the severe deterioration from conditions 5 and 9.

The information determined from the results of testing PU-4 showed that the extraction process occurred to certain extent but was greatly enhanced by the extraction taking place at an elevated temperature. This would indicate that for laboratory testing, where long periods of time are needed to be duplicated in short time spans, testing the elastomer at an elevated temperature is necessary.

#### SECTION IV. CONCLUSIONS

Based on the test results, an extraction process is taking place whereby the protective ingredients placed in an ester urethane are being removed by fuels and the elastomer is undergoing hydrolysis. Compound PU-1 showed that ester urethanes are not resistant to water attack and that some protection is needed. Compounds PU-2 and PU-3 showed that protective ingredients could prolong the life of the urethane but, when those protective ingredients are removed, deterioration still occurs. Compounds PU-3 and PU-4 showed that extracting the protective ingredients at an elevated temperature produced very rapid deterioration.

The reason that diesel fuel is the worst case in the field lies in the fact that it has a low evaporation rate. When diesel fuel is spilled on a collapsible fuel tank, such as during filling procedures, or seeps to the surface from small pinholes, etc., it takes time for it to evaporate or get washed away by rain. During this time, the diesel fuel is able to extract the protective ingredients out of the elastomer allowing hydrolysis to occur prematurely. Other fuels such as gasoline and JP fuels evaporate rapidly thus not allowing the time necessary to extract the ingredients from the urethane.

Additional testing needs to be done to determine standard lest procedures to perform accelerated aging tests on urethanes that are to be used in collapsible fuel storage tanks to assure adequate resistance to hydrolysis even after fuel extraction.

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Figure 1. 5,000 Barrel Collapsible Bulk Fuel Storage Tank Undergoing Testing in Panama



Figure 2. 5,000 Barrel Collapsible Bulk Fuel Storage Tank Undergoing Testing in Panama.

Some Deterioration Visible

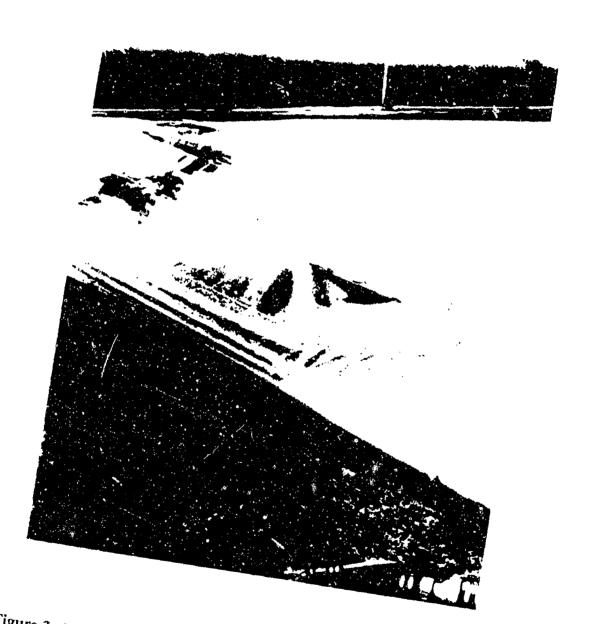


Figure 3. 5,000 Barrel Collapsible Fuel Tank with Seam Failure – View 1

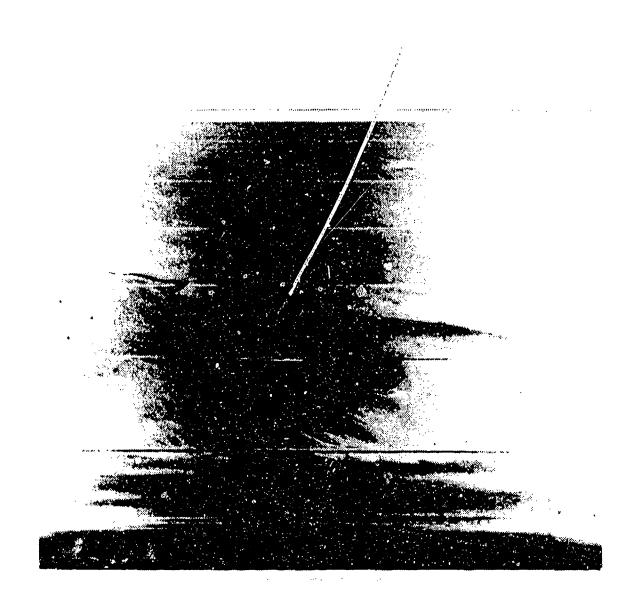


Figure 4. 5,000 Barrel Collapsible Fuel Tank with Seam Failure – View 2

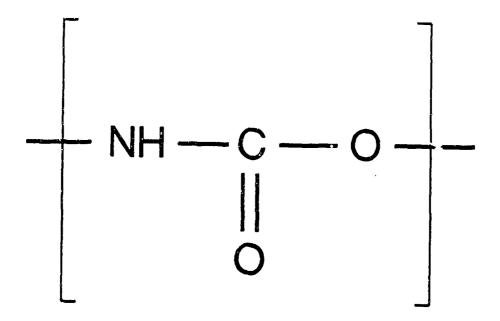
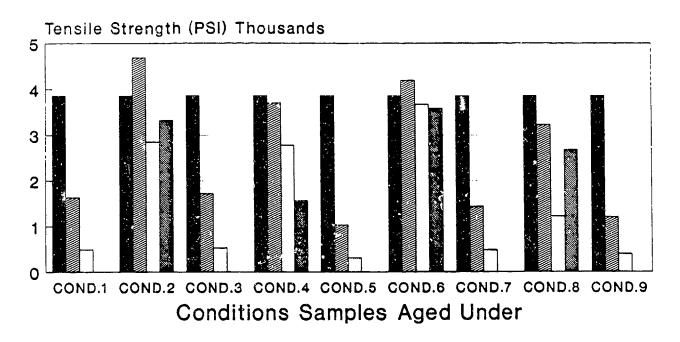
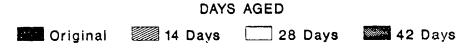


Figure 5. Chemical Description of Urethane Linkage

# POLYURETHANE EXTRACTION TESTING PU-1 TENSILE STRENGTH

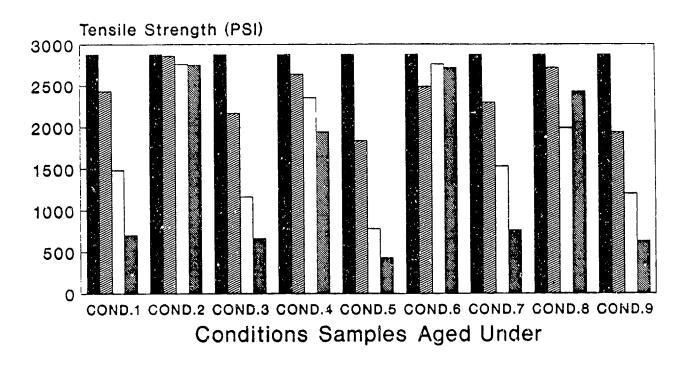




42 Day Samples Failed in Cond. 1,3,5,7,& 9.

Figure 6. PU-1 Tensile Strength

## POLYURETHANE EXTRACTION TESTING PU-2 TENSILE STRENGTH

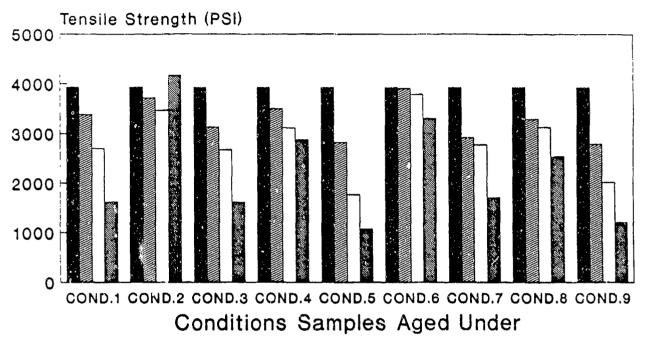


DAYS AGED
Original 14 Days 28 Days 42 Days

MFLL\RCFRG

Figure 7. PU-2 Tensile Strength

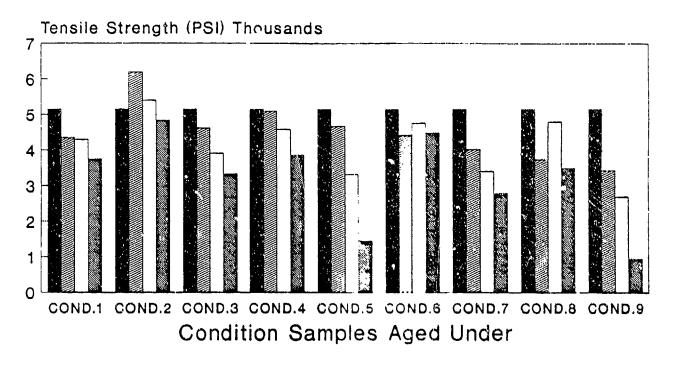
## POLYURETHANE EXTRACTION TESTING PU-3 TENSILE STRENGTH



DAYS AGED
Original 14 Days 28 Days 42 Days

Figure 8. PU-3 Tensile Strength

# POLYURETHANE EXTRACTION TESTING PU-4 TENSILE STRENGTH

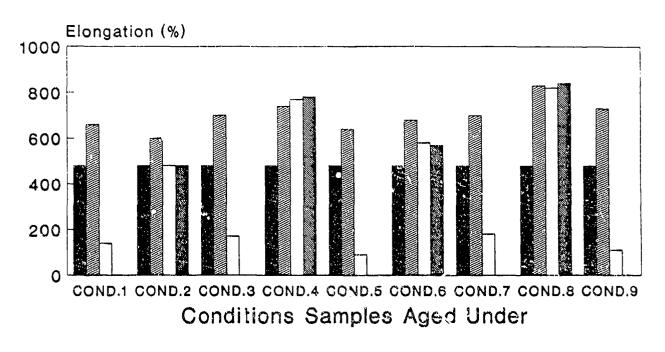


DAYS AGED
Original 14 Days 28 Days 42 Days

MFLL\RCFG

Figure 9. PU-4 Tensile Strength

## PU-1 ULTIMATE ELONGATION PU-1 VLTIMATE ELONGATION



DAYS AGED
Original 14 Days 28 Days 42 Days

42 Day Samples Failed Cond. 1,3,5,7,& 9.

Figure 10. PU-1 Ultimate Elougation

# POLYURETHANE EXTRACTION TESTING PU-2 ULTIMATE ELONGATION

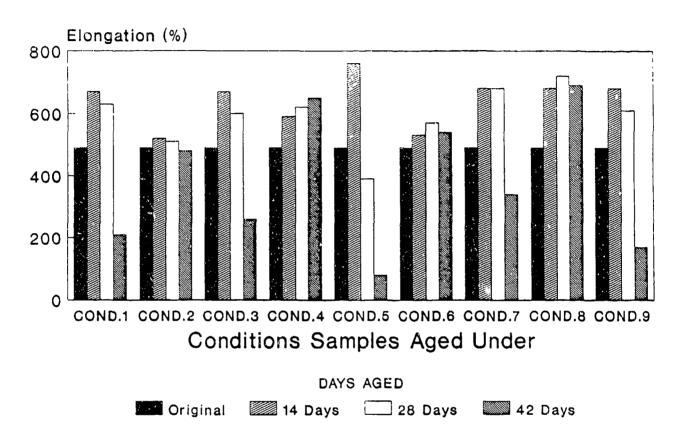
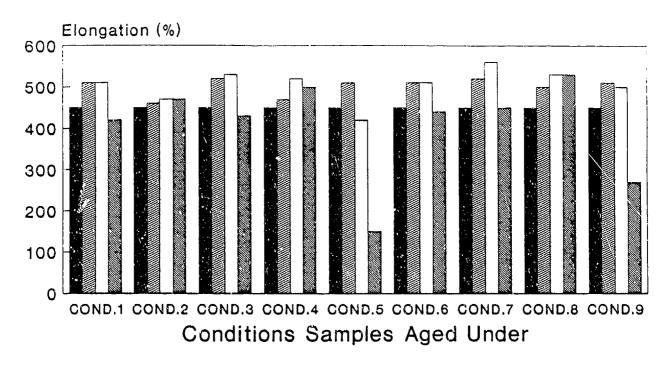


Figure 11. PU-2 Ultimate Elongation

## POLYURETHANE EXTRACTION TESTING PU-3 ULTIMATE ELONGATION

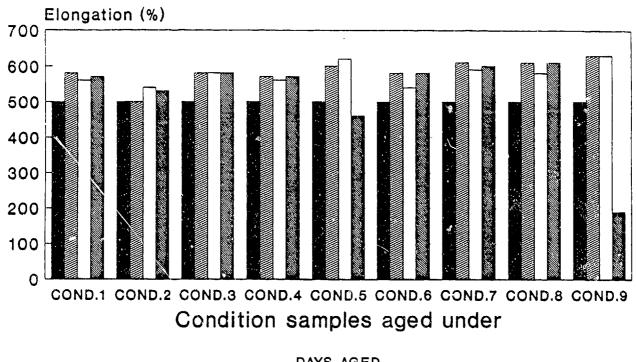


DAYS AGED

Original 14 Days 28 Days 42 Days

Figure 12. PU-3 Ultimate Elongation

# POLYURETHANE EXTRACTION TESTING PU-4 ULTIMATE ELONGATION

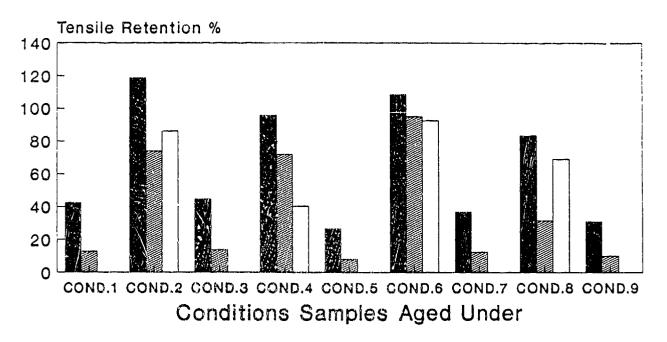


DAYS AGED
Original 14 Days 28 Days 42 Days

MFLL\RCFG

Figure 13. PU-4 Ultimate Elongation

# POLYURETHANE EXTRACTION TESTING PU-1 TENSILE STRENGTH RETENTION



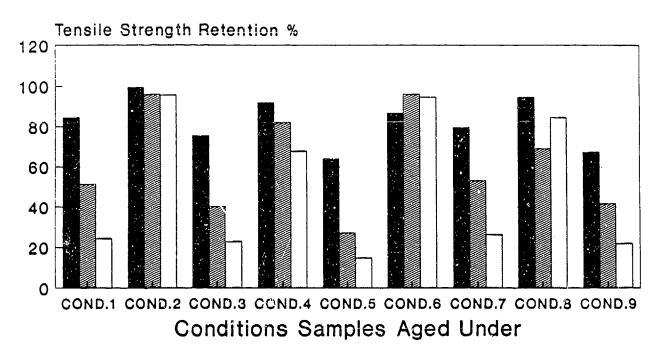
DAYS AGED

14 Days 28 Days 42 Days

42 Day Samples Failed in Cond. 1, 3, 5, 7 & 9

Figure 14. PU-1 Tensile Retention

## POLYURETHANE EXTRACTION TESTING PU-2 TENSILE STRENGTH RETENTION

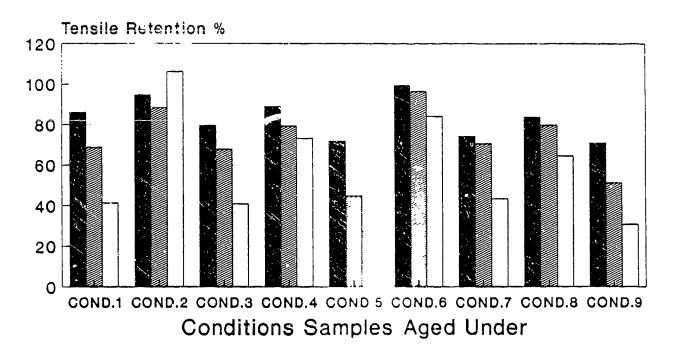


DAYS AGED

14 Days 28 Days 42 Days

Figure 15. PU-2 Tensile Retention

# POLYURETHANE EXTRACTION TESTING PU-3 TENSILE STRENGTH RETENTION

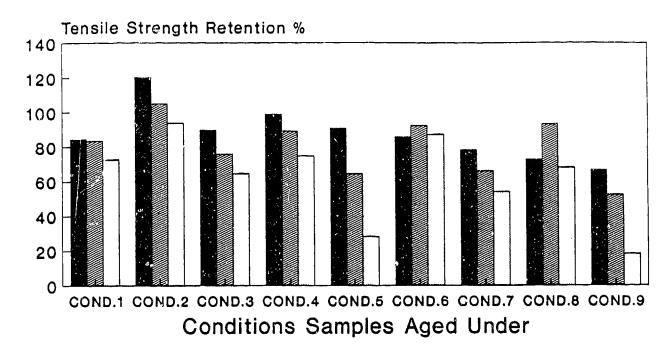


DAYS AGED

14 Days 28 Days 342 Days

Figure 16. PU-3 Tensile Retention

## POLYURETHANE EXTRACTION TESTING PU-4 TENSILE STRENGTH RETENTION

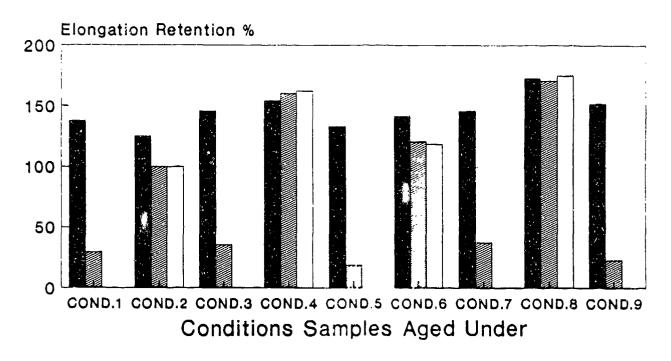


DAYS AGED

14 Days 28 Days 42 Days

Figure 17. PU-4 Tensile Retention

# POLYURETHANE EXTRACTION TESTING PU-1 ULTIMATE ELONGATION RETENTION

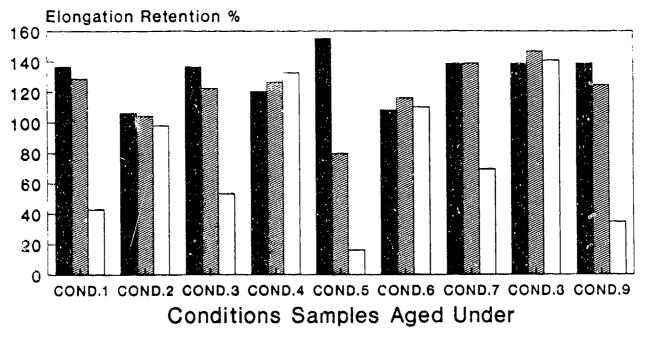


DAYS AGED

14 Days 28 Days 42 Days

Figure 18. PU- "longation Retention

## POLYURETHANE EXTRACTION TESTING PU-2 ULTIMATE ELONGATION RETENTION

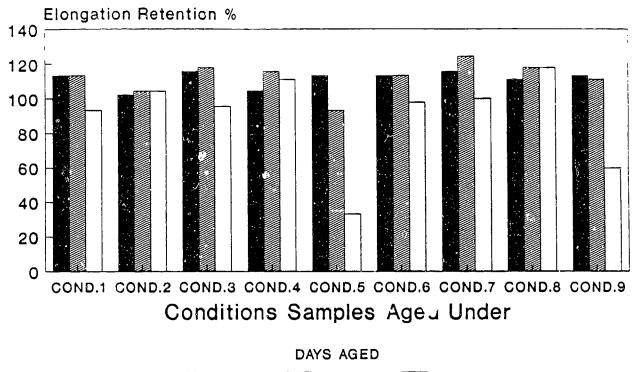


DAYS AGED

14 Days 28 Days 42 Days

Figure 19. PU-2 Elongation Retention

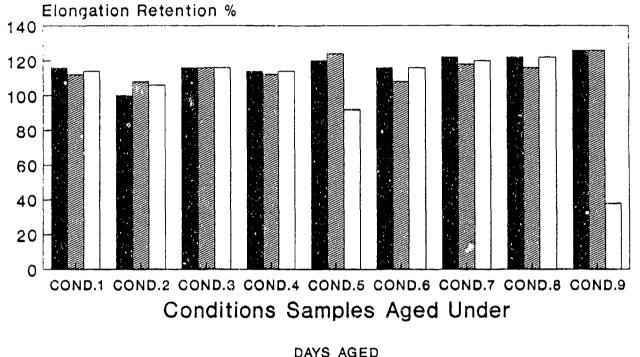
## POLYURETHANE EXTRACTION TESTING PU-3 ULTIMATE ELONGATION RETENTION



28 Days 42 Days 14 Days

Figure 20. PU-3 Elongation Retention

## POLYURETHANE EXTRACTION TESTING PU-4 ULTIMATE ELONGATION RETENTION



DAYS AGED

14 Days 28 Days 42 Days

Figure 21. PU-4 Elongation Retention

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